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Tailoring the mechanical properties of steel sheets using FeC films and diffusion annealing

Elisa Cantergiani [a], Amélie Fillon [b,1], Ben Lawrence [c], Xavier Sauvage [b], Michel Perez [d], Colin P. Scott [e], Arnaud Weck [a,f,g,*]

[a] Department of Mechanical Engineering, University of Ottawa, 161 Louis Pasteur, Ottawa, ON, Canada K1N 6N5
[b] Université de Rouen, Groupe de Physique des Matériaux, UMR CNRS 6634, BP-12, 76801, Saint Etienne du Rouvray Cedex, France
[c] The University of British Columbia, Dept. of Materials Engineering, 309-6350 Stores Road, Vancouver, Canada V6T1Z4
[d] Université de Lyon, MATEIS-INSA-Lyon, UMR CNRS 5510, 69621 Villeurbanne, France
[e] Canmet MATERIALS, Hamilton, ON, Canada L8P0A5
[f] Department of Physics, University of Ottawa, 150 Louis Pasteur, Ottawa, ON, Canada K1N 6N5
[g] Centre for Research in Photonics at the University of Ottawa, 800 King Edward Ave., Ottawa, ON, Canada K1N 6N5

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ABSTRACT

In this work amorphous FeC films were deposited on thin sheets of interstitial free steel using physical vapor deposition. Annealing treatments were then carried out to diffuse C from the coating into the substrate at temperatures lower than those traditionally used in carburizing treatments. The yield stress was shown to significantly increase with annealing temperature from ~120 MPa at 25 °C up to a maximum of 300 MPa at 630 °C without any significant loss of ductility. At 710 °C, a decrease in yield strength was related to the coarsening of carbides inside the IF steel (confirmed by atom probe tomography), and the associated reduction in the matrix solid solution carbon concentration (confirmed by thermoelectric power measurements). The through-thickness carbon diffusion profile was predicted using Fick's law and validated by Knoop hardness measurements. Yield strength predictions were accurate if the crystallization of the FeC film was taken into account as it controls the amount of carbon available to be diffused in the interstitial free steel substrate.

1. Introduction

Traditional techniques used to strengthen steel by carbon additions involve case hardening and bake-hardening. Case hardening is related to the diffusion of carbon in bulk materials to increase their surface hardness and strength and it includes many techniques such as: pack carburizing, gas or liquid carburizing and vacuum carburizing. All these processes require temperatures higher than 870 °C and hours of processing times [1–3]. These processes are generally used to case-harden the surface of thick materials and cannot easily be applied to harden thin sheets of steel [1]. The traditional process used to strengthen thin sheets of ultra-low carbon steels is bake-hardening. This heat treatment (170 °C for 20 min) is performed after the forming step in order to segregate free carbon to the dislocations. However, the increase of yield stress caused by this heat treatment is limited to +50–60 MPa because the amount of carbon initially present in solution in the steel has to be kept low enough to avoid room temperature ageing which would cause Lüder's instabilities to appear during forming [2]. Steel companies are interested in developing new methods which can further increase the yield stress of thin sheets, without compromising the ductility or processing capability. The new approach proposed here is to use an iron–carbon film deposited on the surface of a steel sheet by Physical Vapor Deposition (PVD). These films, which can be crystalline or amorphous, have a very high carbon content and therefore can act as carbon reservoirs during subsequent diffusion annealing [4,5]. The advantage of the iron–carbon system is that the adhesion with the IF substrate is excellent and the corrosion resistance is improved. PVD is already implemented in the steel industry [6] and can be used as a continuous processing technique to coat steel strips wider than 1.5 m [4]. The present scope is to diffuse carbon at temperatures lower than 800 °C with a more time efficient process than the traditional carburizing procedures. Depending on the annealing temperature and time the final sheet can have a gradient of carbon or a uniform distribution of carbon through thickness. Preliminary work on this technique was made by Scott et al. [4], where it was shown that these films can be used to

* Corresponding author at: Department of Mechanical Engineering, University of Ottawa, 161 Louis Pasteur, Ottawa, ON, Canada K1N 6N5.
E-mail address: aweck@uottawa.ca (A. Weck).
[1] Present address: INSA Rennes, ISCR UMR CNRS 6226, Laboratoire Chimie-Métallurgie, 20 Avenue des Buttes de Coësmes, 35708 Rennes, France.

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strengthen a sheet of interstitial free steel. In the present work a detailed investigation of the influence of film thickness, annealing temperature, and annealing atmosphere on the diffusion process and on the related mechanical properties is presented.

2. Experimental procedure

2.1. Material and physical vapor deposition

The substrate material is a fully recrystallized 0.197–0.200 mm thick Interstitial Free (IF) steel whose composition is shown in Table 1.

The material is largely over-stoichiometric in Ti in order to completely stabilize the C and N in the form of TiC and TiN precipitates. Dog-bone shaped tensile coupons of IF steel with a gauge length of 5 mm, a width of 1 mm, and an average thickness of 0.197 mm were cut using electro discharge machining (EDM). Tensile coupons were then cleaned on both sides using ethanol followed by a 2 vol% Nital solution, and then rinsed using distilled water and ethanol. After the cleaning procedure, specimens were coated on both sides with an amorphous carbon-containing film (FeC) using physical vapor deposition (PVD). In addition to the deposition parameters already detailed in [4], pulses of methane were injected in the chamber to dynamically control the voltage between target and substrate which in turn controlled the carbon content in the film. The final carbon amount in the FeC film was 30 at% measured by Auger electron spectroscopy (AES) and by Atom Probe Tomography. At the end of the PVD process, 500 nm thick film were deposited on both sides of the IF steel substrate.

Another set of experiments was performed to investigate the influence of film thickness on carbon diffusion. Tensile coupons were coated on both sides using the PVD procedure illustrated above, with 50 nm, 100 nm, 200 nm, 300 nm and 400 nm thick FeC films.

2.2. Heat treatment

To investigate the diffusion of carbon from the film into the steel substrate, annealing treatments were carried out at temperatures of 330 °C, 430 °C, 530 °C, 630 °C, and 710 °C for 1 h in high vacuum (average pressure of 6×10^{-4} Pa). Specimens were heated inside a vacuum furnace at a heating rate of 12 °C/min until the target temperature was reached and held at that temperature for 1 h. The specimen was left to cool down inside the furnace with an initial cooling rate of 12 °C/min when the temperature was above 400 °C and then at 3 °C/min when the temperature dropped below 400 °C. Samples used to investigate the effect of film thicknesses were annealed at 530 °C for 1 h under high vacuum (average pressure 6×10^{-4} Pa).

To study the influence of the annealing environment, a series of tensile coupons were annealed for 1 h in an argon atmosphere at 530 °C and 630 °C, and others in air at 530 °C.

2.3. Mechanical properties

Tensile tests were carried out at a strain rate of $5\times10^{-4}\ s^{-1}$ on a minimum of three annealed coupons for a given temperature. For each test, yield stress, ultimate tensile strength (UTS), strain at fracture, Lüder's strain and Lüder's plateau length were obtained. For specimens showing Lüdering, the yield stress was taken as the upper yield stress, while for coupons without a plateau, the yield stress was taken as the stress at 0.2% engineering strain. The fracture surface of the tensile coupons was analyzed using scanning electron microscopy and the reduction in cross sectional area was calculated using the open-source image analysis software ImageJ [7].

Knoop hardness was measured to estimate the shape of the through-thickness carbon profile for the whole set of annealing temperatures (430 °C, 530 °C, 630 °C and 710 °C for 1 h). Knoop microhardness was preferred to Vickers since the depth of the indent is smaller for the same loading condition, which allowed for a better spatial resolution of the carbon profile. Knoop hardness was measured by applying a load of 100 g for 10 s. Before each hardness measurement, the specimen was polished to remove 20 μm, out of which 10 μm were removed using 4000 grit SiC paper, while the remaining 10 μm were removed using 3 μm, 1 μm and 0.05 μm polishing solutions. This procedure ensures no influence of polishing on the hardness results. After 20 μm were removed, Knoop hardness was performed again. The procedure was repeated until the center of the sheet was reached. For each hardness point, 15 Knoop measurements were recorded and the average hardness value was calculated discarding the two most extreme data points.

Table 1
IF steel composition.

	C	Ti	P	Mn	S	N
$\times10^{-3}$ at%	14	64	16	113	22	7

2.4. Transmission electron microscopy (TEM)

The microstructure of the coated IF coupon was characterized by Transmission Electron Microscopy (TEM) using an ARM200F JEOL microscope operated at 200 kV. Cross-section TEM samples were prepared using a focused ion beam (FIB; NVISION-40 Zeiss).

2.5. Atom probe tomography (APT)

Local composition measurements of the substrate material were carried out by atom probe tomography (APT) on samples prepared by FIB milling. Analyses were performed in ultra-high vacuum conditions, using an energy-compensated atom probe (EcoTAP CAMECA). Samples were field-evaporated using electric pulses (repetition rate 30 kHz and 20% pulse fraction) at 80 K. Six samples were selected for APT measurements. One uncoated and not annealed sample, two samples coated and annealed at 530 °C for 1 h and three samples coated and annealed at 710 °C for 1 h. For the annealed samples, one APT specimen was extracted from the middle of the steel substrate while the others were taken close to the film/substrate interface.

2.6. Thermo electric power (TEP)

To evaluate the amount of interstitial carbon in the samples after heat treatment, thermoelectric power measurements (TEP) were carried out. The set-up used for TEP measurements has been already described in the literature [8,9], and the protocol used for the present analyses was explained in detail by Lavaire et al. [10]. The protocol comprises a cold-rolling step (70%), which is performed to introduce a large amount of dislocations. Then, the TEP value of this cold-rolled state is assessed and the material is then aged (120 °C for 30 min) causing the segregation of all interstitial atoms at dislocations. After the ageing treatment, the TEP value is assessed again and the difference between this value and the value found after cold-rolling is related to the concentration of interstitial elements. TEP tests were carried out on coated IF steel after annealing for 1 h at 430 °C, 530 °C, 630 °C and 710 °C. For comparison, the same tests were also performed on uncoated IF steel.